

INSTRUMENT OPERATING PROCEDURE

INSTRUMENT TYPE: Total Organic Carbon (TOC) Analyzer

MANUFACTURER: Rosemount Analytical, Dohrmann Division

MODEL: DC 190

SERIAL NO.:
DC 190 Electronics Module Serial No. 96274012
DC 190 IR Detector Module Serial No. N6H3681C
DC 183 Sludge/Sediment Sampler Serial No. 96348002

DEPARTMENT: Inorganics - Wet Chemistry

PROCEDURE:

The DC 190 TOC Analyzer can be configured several ways. This procedure will explain the configuration for the analysis of waters (WTOC) and soils (STOC) and will be dealt with in separate sections.

I. Waters TOC (WTOC) configuration

A. Routine Operation

1. The unit will be in a stand down mode prior to start up. The unit and the detector are always "on".
2. The fan for the detector and the combustion tube, as well as the one in the soils unit, should always be "on".
NOTE: Do not touch any of the buttons or knobs on the detector !
3. Check the waste drainage port. The waste tray should be emptied prior to and during operation (if it becomes full).
4. Check the volume of acid in the amber bottle and add if volume is 1/2 or less. (20% phosphoric acid.)
5. Check that all tubing from the previous run has been reconnected (needed only after soils runs).
6. Remove any water or moisture from the water trap (inside the unit).
7. The main screen should read:
 1. System Status
 2. Auxiliary FunctionsPress # 2, it will read # 1:Omit outliners, etc.

- Press # 5, Service Functions, it will read # 1: Prime acid.
- Press # 1 and it will read # 1: Acid to IC Chamber.
- Press # 1 and it will add acid. When it is finished, return to the main menu by pressing MAIN.

Press # 1 for System Status. Flow rate # 1 and furnace set point # 4 will be shown. Turn on O₂ valves in this order ONLY: Open the tank valve, then SLOWLY open the regulator valve. Doing the reverse may blow out the valves!

The display pad has two buttons - one labelled Furnace and the other Carrier gas. Press the Carrier gas button. The light will change from red to green. Press the furnace button. The light will change from red to green when the furnace temperature has reached the set point. Analysis cannot begin until the set furnace temperature has been reached. The temperature will be 550 °C at stand down, and should be set at 680 °C for analysis.

The flow rate should be adjusted to 200 mL/min \pm 20 %. This can be adjusted at the regulator.

8. Change the rinse water container DAILY. This should be acidified Milli-Q water. The sparge arm assembly can be manually moved out of the way (to remove the sample tray). The sample arm will not move manually. Follow the flow diagrams on pg. 2-19 of the manual to move the sample arm.
9. Check PEEK tubing (the beige tube on sample arm and sparge arm) to be sure it is clean and straight.
10. Observe the baseline (bottom right corner of display screen).
11. The unit is now ready to start analysis for all aqueous modes. Except POC: lithium hydroxide must be added to the scrubber tube seen as # 7 on pg. 2-7.

B. For ASM/NPOC:

1. Set parameters prior to beginning analysis. Check parameters even if the previous run was similar. When analysis is started, the parameters are locked in. To change them after analysis has begun, press start/stop 5 times to terminate the run.

Press the NPOC button. A list of actions will appear.

- Injection volume
- Number of repeats
- Sparge time
- Acid volume
- Rinse or stir
- Print set up

- a. Injection Volume can range from 10 μL - 400 μL . For most water, it should be set to 400 μL . If the autorange is on, this volume will change according to what the TOC reads. To change the volume prior to analysis, press # 1 to highlight the volume to the right. Press Clear. Type in the desired volume and press Enter.
- b. Number of Repeats corresponds to the number replicates of a single vial. The default is three for all vials to be read.
- c. Spurge Time refers to how long a sample will be sparged with O_2 . The default is three minutes.
- d. Acid Volume corresponds to the amount of acid added when sparging: one pulse is equal to 100 μL .
- e. Rinse or Stir refers to another menu, which lists the number of rinses with water, the number of rinses with sample, stir time (stirring prior to each aliquot of sample or standard), autorange (yes or no), and CG on or off at the end of a run.

If there are any changes made to this set up, it should be noted prior to the run and noted in the run log.

2. When beginning a first run for the day, determine a new calibration factor and system blank. After these have been set, any number of sets of 32 vials can be run with that calibration for that day. The same calibration may be used on consecutive days as long as the calibration check standards pass to demonstrate the instrument is still in calibration..

Press the calibration button. A list of actions will appear.

- The calibration factor shown will be from the previous day's set up.
- The system blank shown will be from the previous day's set up.
- The sample size as set from NPOC mode.
- Standard concentration
- Update calibration factor
- Update system blank
- Other actions

Press # 1 and the value will be highlighted. Press clear, type in 1.0, press Enter.

Press # 2. Press clear, type 0, press Enter.

4: Make sure this is the value that will be read.

(Numbers 5 and 6 are used when in Boat/Soils mode.)

3. Note the baseline on the screen (bottom right corner). It should be steady and not drifting up or down. Once a run has started, the unit will return to the memorized baseline. If the baseline is unsteady during the run, the results will also show drift,

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or there will be a time out error. Check the manual's troubleshooting section if this occurs.

C. Begin analysis

1. Sample vials should be 1/2 to 2/3 full. Any more than this will result in carryover from one vial to another via the orange rubber septum.
2. Place the black tray onto its peg, line up the silver push peg with its slot on the tray. Try not to force the tray to turn, it has memory regarding each vial position. Make sure samples and standards are well mixed prior to aliquoting.

D. Calibration

Check manual for method-specific calibration.

To calibrate ASM/NPOC : check parameters.

In vial # 1, place the standard

In vial # 2, place the same standard with a peg on the outside of the circle.

In vial # 3, place Milli-Q water

In vial # 4, place Milli-Q with a peg on the inside of the circle

The unit will automatically perform the calibrations. Follow with calibration checks and LCS or samples. A CCV/CCB set should follow every 10 vials. Keep track of vials on the run log. To terminate the analysis, place a peg on the outside circle next to the last vial to be read.

If at any point the analysis needs to be terminated, press the start/stop button 5 times.

When the analysis is complete, send the information to the computer and printer.

Another method can now begin or the unit can be shut down.

E. Shutdown

Leave the furnace "on" at all times, the exception being if it is not to be used for several weeks. If it is not to be used for several days, go into the system menu (from the main menu) and change the set temperature for the furnace to 550 °C.

Turn the carrier gas off. Turn the regulator valve off, although the tank can be left on.

Empty vials. Dump the waste tray.

DO NOT turn either the detector or the unit itself off.

II. Soils TOC (STOC) Configuration

A. Routine Operation

Beginning with the unit in a shut down mode:

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1. Open the door and take the teflon tube out of the copper side of the tin/copper scrubber. Note its position prior to removing (ie., how much tube is actually inside the U tube). Connect the tube labelled soils (with black shrink tubing around it) and connect it to the tube from the copper side.
2. Place the tube and septa into the sparger on the soils unit.
NOTE: Remove after analyzing soils - H₂O will travel back into the furnace if this is not done.
3. Turn boat gas and sparge gas on (remember to turn off when done!).
4. Turn power on (black switch). Indicator light shows when it has warmed up.
5. Turn the carrier gas on at the tank, then at the regulator. In the NPOC/Boat mode, type in the concentration units: (µg/g or mg/L). For solids (soils), use µg/g. Sample size indicates size of standards to use. Analysis can now begin.

NOTE: In this mode, after reading the boat, the unit will ask if you want to continue (meaning: Do you want to perform more than one replicate?). Generally, as with waters, a sample or standard can be read in triplicate and the first value omitted. In all modes but the ASM/RSM, this can be done by the TOC itself. See Omitting an outliner.

6. Prepare samples as described in the SOP for soils. A homogeneous mix should be achieved. Have the sample or the standard ready to inject: that is, weighed or aliquoted, in the boat, the boat in the carrier with the lid closed, prior to pressing the Start button.

B. Calibration:

1. Select the calibration function and change the necessary items (ie., calibration standard concentration).
2. In order to achieve a mean value for any standard, perform at least three injections. After this, the unit asks to continue or not, press no. The unit will perform the calculations. Omit any numbers, if needed, at this time.
3. Select the calibration function and update the calibration factor. Since there is no blank to be standardized, a system blank need not be updated.
4. Calibrate with the standards used in the SOP for the Boat mode. When complete, the unit is ready to read samples. Apply the same procedure for the soil samples. Load the boat and place in the slide. Press Start.
5. Follow the guidelines for MS/MSD and other qualifications as stated in the SOP for soils analysis.

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C. Shut down

1. Remove boat from slide.
2. Turn the boat gas and sparge gas off. Turn the carrier gas off if no further analysis of waters is needed. Turn both the tank and the regulator off.
3. Remove the tube from the gas sparger tube on the soils unit.
4. Turn the power off on the soils unit (black switch).
5. Open the door of the autosampler unit and remove the tube from the outer black shrink tubing. Place the tube (without black tubing) into the copper side of the tin/copper scrubber. Return the tube in the copper scrubber to its position prior to removal. This is important for gas circulation in the tube.
6. The fan for the soils unit will always be on in order to cool the furnace.

III. Routine/Preventative Maintenance

Check the manual for troubleshooting and any routine maintenance.

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